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2) Hughes, Ronald Clyde
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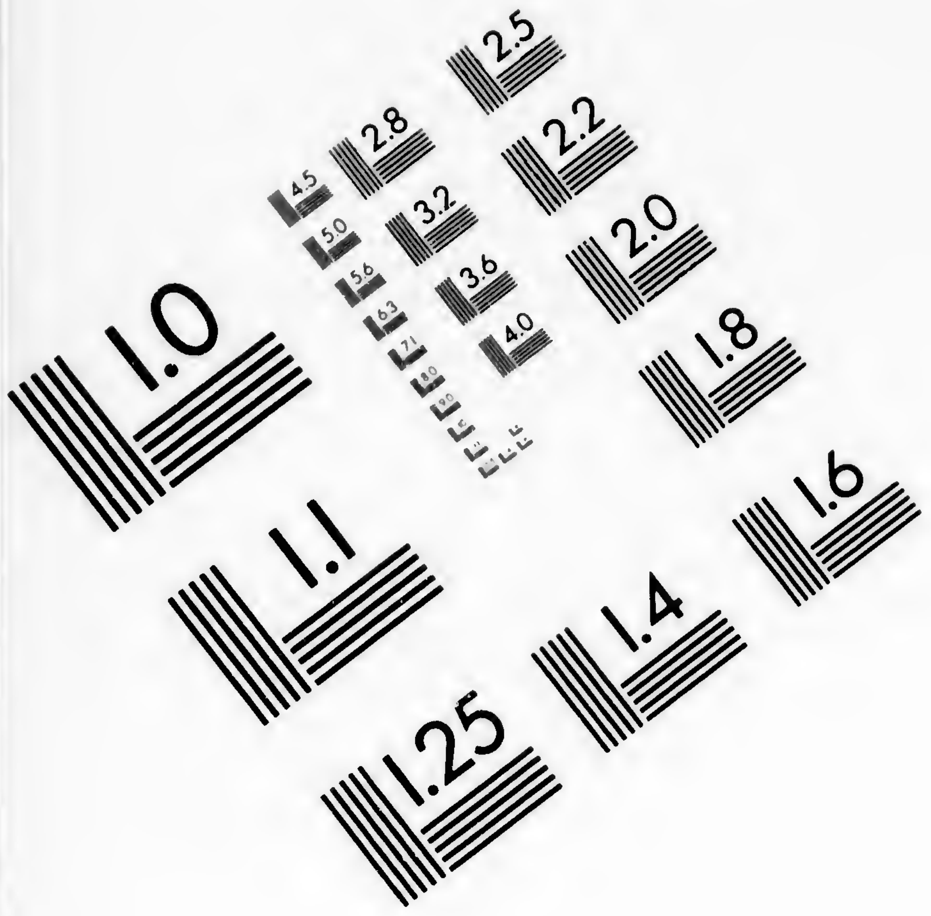

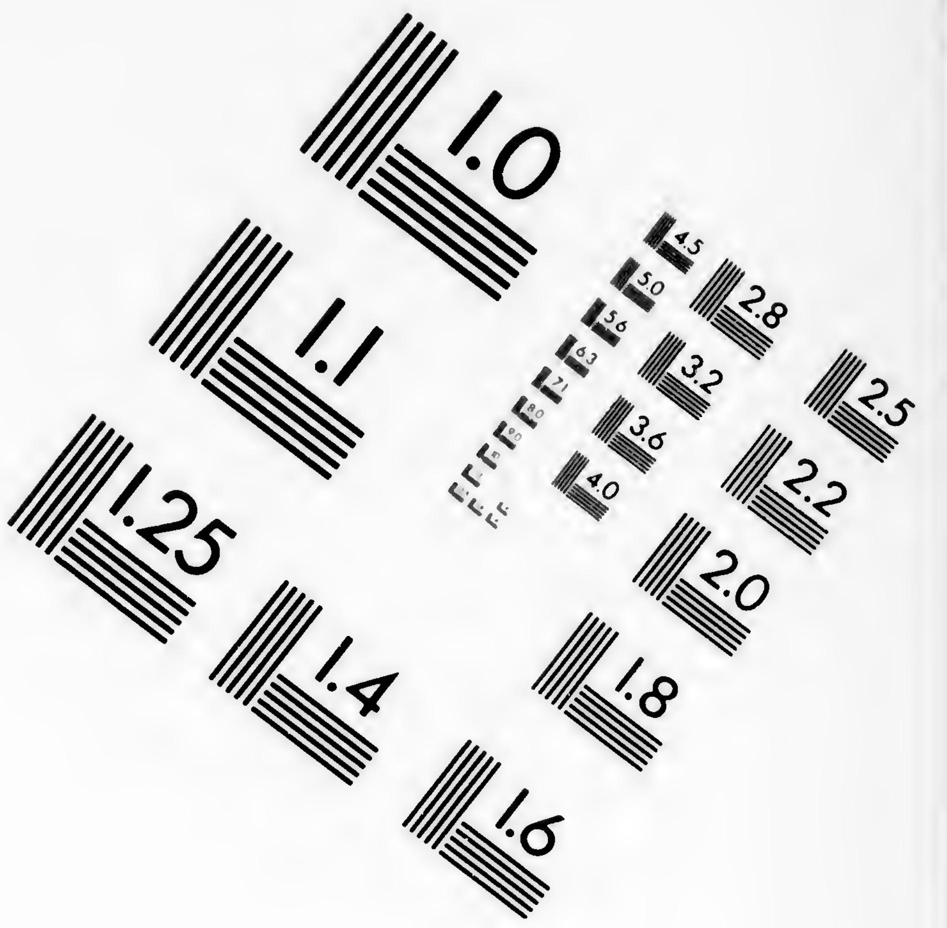

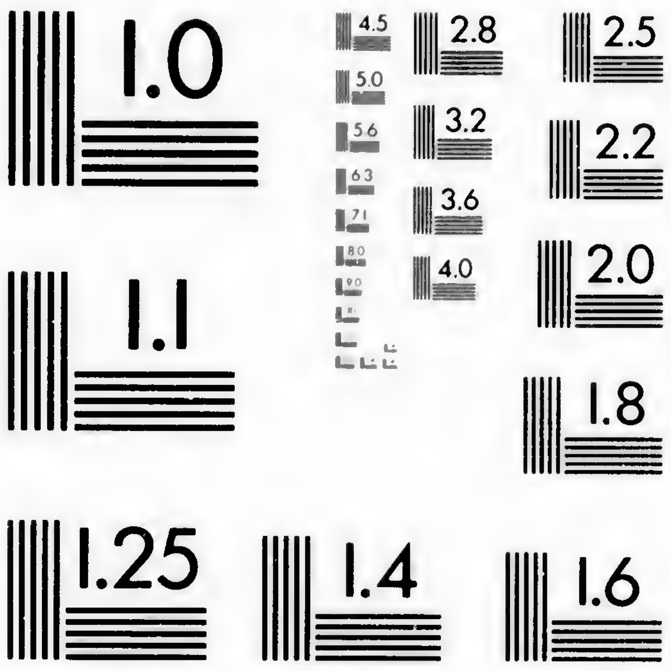

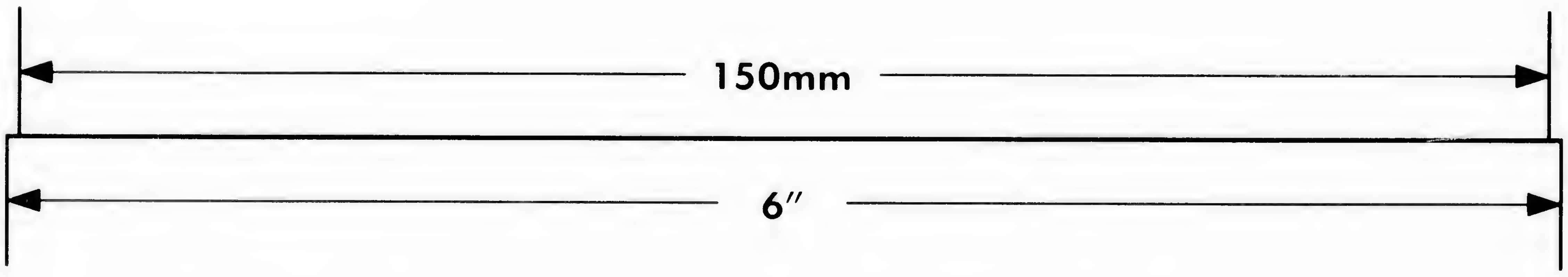

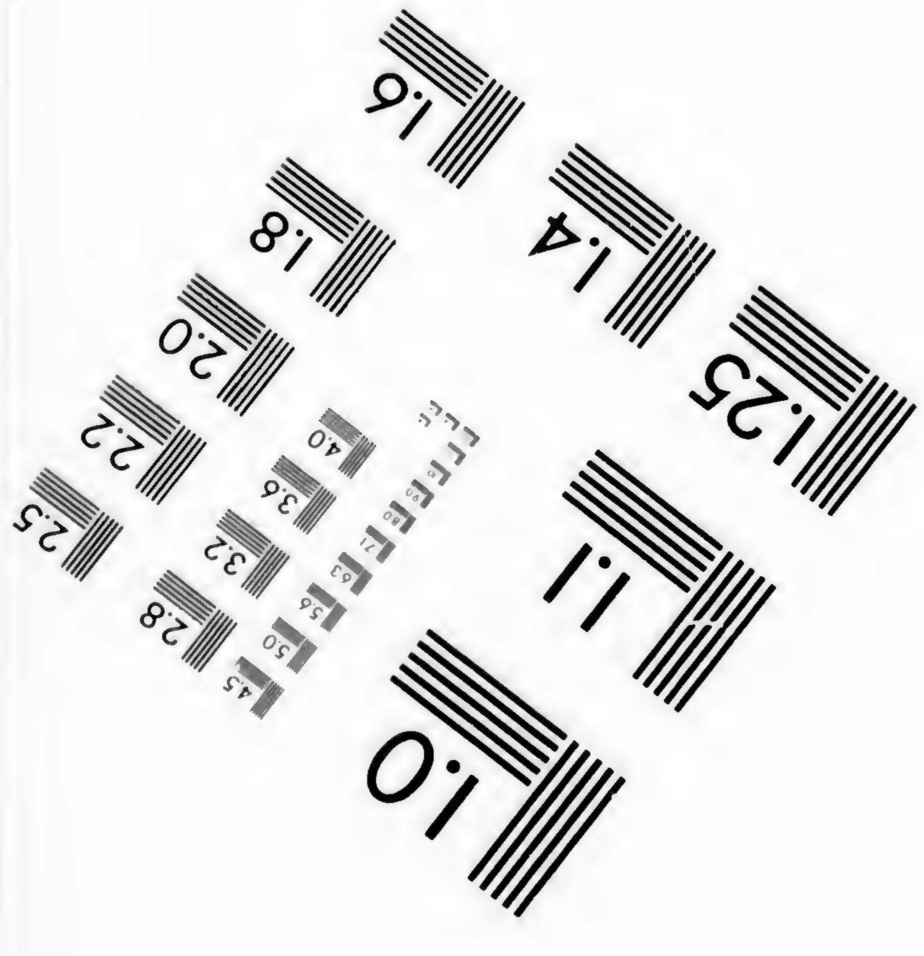

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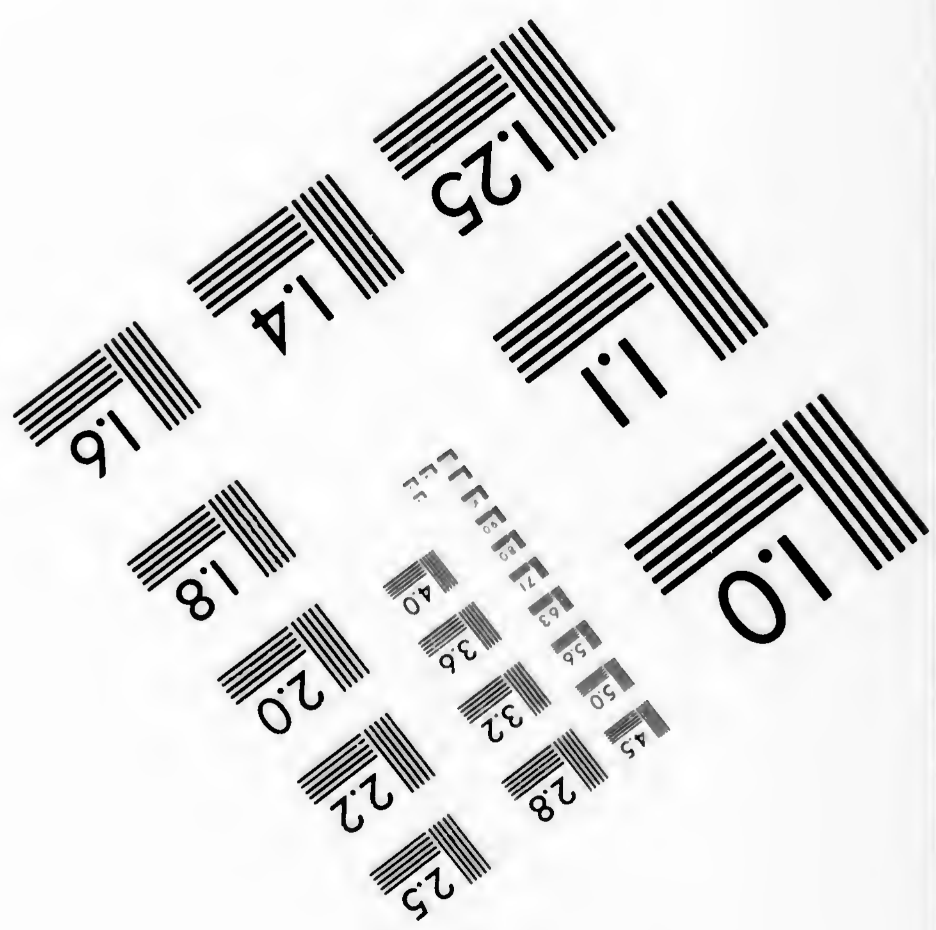

THESIS

# PHYSICO-CHEMICAL FACTORS AFFECTING THE COMBUSTION OF PENNSYLVANIA CIGAR-LEAF TOBACCO

Submitted to the Committee
on Graduate Study and Advanced Degrees
of The Pennsylvania State College

by

Ellis Philip Baker
in partial fulfillment of the requirements
for the degree of
Master of Science
1928

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Approved May 29 1928

R. Adams Dutcher
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# PHYSICO-CHEMICAL FACTORS AFFECTING THE COMBUSTION OF PENNSYLVANIA CIGAR-LEAF TOBACCO

Of the many criteria by which good cigar tobacco is judged, no single one is more important than the character of the "burn", or "burning quality" as it is generally termed.

There is a very wide variation in cigar-leaf tobaccos as pertains to the burning qualities, and it appears from the research in this field that the cause for this variation lies in the differences in chemical make-up of the leaves, which in turn is dependent to a great extent upon the present availability and of the certain elements in the soil necessary to catalyze the combustion and the lack of substances which interfere with this process in the finished commercial product.

It may be well to point out that combustion spoken of in the sense here does not mean combustion in the gaseous sense, rather it means an incandescent combustion or glow; thus fire-holding capacity can be qualified as the ability to maintain a temperature high enough to support incandescence of the mineral elements present. It is known from experimentation that this degree of temperature can be more readily obtained by speeding up the reaction velocity of the combustible materials by the addition of certain catalyzers.

Many research workers have focussed their attention upon determining the chemical constituents necessary to make for good burning qualities and have deduced from their results certain explanations for the apparent relationship of these constituents to a good burn.

In the past, two methods of attack have been used, one analytical, the other synthetic.

The analytical methods depend upon the identification and character of the chemical compounds present in the leaf.

The synthetic method consisted in adding to the tobacco various chemical compounds which normally occur in the leaf, and studying their effect upon the burn.

## Review of the Literature

The initial investigation begun in this field was performed by Schlösing(15) who found that potash in contact with organic acids was favorable to the fire-holding capacity, but potash combined with the mineral acids, sulfuric and hydrochloric, inhibited the burn. He attributed the favorable action as due to the swelling of the potash salts, resulting in great porosity which gave finely divided carbon when heated.

Nessler(13) tested the effect of various salts on the combustion of filter paper and concluded from his results that potash in the form of the sulfate and carbonate were very favorable to the burn, chlorides were very injurious, lime and magnesium whitened the ash considerably and the nitrate of potash gives a very quick but incomplete burn. He discredited Schlösing's theory, however, because not all the salts found favorable to the process had the property of swelling when heated.

Mayer(14) carried on some experiments similar to those of Nessler, but included organic compounds, and found that they favored burning with a flame, but, in general, did not improve the burning capacity.

Conclusive results cannot be formed from these experiments because, as Garner(9) points out, the results found on filter paper may not be true of tobacco because of the difference in the chemical make-up and structure of the two substances.

It has been found that in good burning samples the potash is largely in excess of the acids while the reverse is true of samples showing a poor burn.

Garner has suggested that this effect might be due to the fact that it yields the carbonate easily, and the carbonate in giving off and taking up $CO_2$ might be favorable to the burn.

Kraybill(17) studied the effect of alkali salts upon the burn. Since lithium carbonate dissociates much more easily than potassium carbonate, one would expect lithium carbonate to be even more effective from the above explanation. However, Kraybill did not find this to be true.

Barth has suggested that the glow of a good tobacco might be due to the reduction of the alkali salts to the oxide and small quantities of free metal which act as oxidation catalysts.

Nessler(13) also thinks that potassium salts are reduced and may act as oxygen carriers.

Garner(9) made water extracts of the leaf and found that the extracted leaf had lost its power to glow. An examination of the extract revealed the fact that it contained the chloride, sulfate, nitrate, malate and citrate of potassium, ammonium and nicotine. Extracts from a leaf showing poor burn were found to contain a higher percentage of mineral acids than the good burning leaf. He concludes from this that the principal factor favoring the burn is the potash in excess of the amount required for combination with the mineral acids present. From this he further concludes that the potash in combination with organic acids is a factor having a favorable effect on the burn.

The rôle that iron plays in biological oxidations cannot be overlooked. As examples can be mentioned: (1) the action of iron and blood in the growth of Bacterium lepisepticum, (2) the use of iron hydroxide in the oxidation of lactic acid to pyruvic acid, and (3) the use of ferrous hydroxide and oxygen in the reduction of potassium nitrate.

In inorganic chemistry, the use of iron as a catalyst is widely spread. In the Claus-Chance process for the fractional combustion of hydrogen sulfide much use is made of hydrated ferric oxide, while in the purification of illuminating gas, precipitated oxide of iron, an artificially prepared material has come into considerable use as a catalytic material. Much use has been made of oxide of iron as a catalyst in the direct synthesis of ammonia.

Almquist and Crittenden(1) investigated the use of iron as a catalyst and found that its efficiency depended upon its state of oxidation, also that the mixture which approached the theoretical ratio $FeO - Fe_2O_3$ was the best and that the activity of this could be increased by the addition of certain other oxides slightly basic in nature.

Armstrong and Hilditch(2) investigated the X-ray photographs of the mixed oxides of Almquist and Crittenden. This experiment brought out some fundamental differences between pure iron and "promoted" iron, i.e. those with basic oxides as "promoters." While the crystals in iron containing no promoter grow rapidly, crystals of iron in the best catalysts grow slowly, therefore a promoter maintains a large surface by preventing a sintering (slipping of the crystal forces) of the iron.

Wolff and de Stecklin(20) found that if any iron salt, such as the nitrate, sulfate or acetate possesses a catalytic effect alone, it can be increased by the addition of an alkaline sulfocyanide.

Baudisch and Welo(4) attribute the action of iron salts as being due to the fact that the molecular oxygen loosely bound to the iron may act as a dehydrogenating agent toward the oxidizable substance present in the mixture. They have shown that the activity depends on the size of the crystals and also on the structure of the crystal.

## Object of the Investigation

The object of this investigation was: (1) to devise a suitable method for estimating the burning quality, (2) by means of this method study some of the chemical factors affecting the burning quality.

## Experimental

### Apparatus and Materials Used

The apparatus used consists of a section of brass gas tubing into which moist tobacco is placed and subjected to a pressure of      dynes by means of a plunger. (See Figure B.).

The compressed tobacco is then gently pushed out of the tube in the form of a pencil (.7 cm. in diameter). These are then placed in a drying oven at a temperature of 40°C. for twenty-four hours.

The samples used for the experiment were made up of a tobacco having a very poor fire-holding capacity. The first set of samples, each sample weighing five grams, was treated with varying quantities of water to determine the quantity most suitable as a cementing agent. It was found that samples treated with an amount of water which equaled 55 per cent of the weight of the sample on the air dry basis gave optimum results.

These samples when dry were put in an especially constructed rack (see Fig. A.). A distance of one inch was measured off on the pencil, a piece of thread treated with a saturated solution of sodium nitrate was placed at the two extremes of this distance, and the sample ignited. Care was taken that the pencil burned without a draft. When the glowing portion reached the thread, a flare resulted. The interval between the flares of the first and second threads was noted and recorded by means of a stop watch. In this way a very accurate check on the relative fire-holding capacity of the various samples was obtained. Samples were made up containing various concentrations of potassium hydroxide, potassium citrate, potassium carbonate, potassium tartrate, potassium nitrate, sulfate and chlorides. Like volumes of these solutions were the same as those used in the case of water as a cementing agent.

Samples were also made up containing various concentrations of ammonium citrate, iron ammonium citrate mixtures of iron acetate and potassium carbonate or citrate; also mixtures of iron acetate and aluminum oxide. The actual concentration of these solutions in terms of normality are given elsewhere.

As a preliminary to the experimental work described, samples of the 1925, 1926 and 1927 Pennsylvania crop were made up and burned by the method outlined, in an effort to determine the relative fire-holding capacity. The results, which were found to bear out previous determinations on the burn of these tobaccos, are given on a following page.

## Presentation of Data

The figures are plotted in the following way. The time is plotted along the ordinate and the concentration along the abscissa. The red line indicates the time of burn for those samples treated with water only. For the sake of clearness this line is designated as the "normal" for the particular type of tobacco which was used.

## Results on the Preliminary Experiments

### Crop of 1925

| Sample | Burn |
|---|---|
| A-1 | Poor |
| A-2 | Poor |
| A-5 | Medium |
| A-6 | Good |
| A-9 | Poor |
| A-10 | Poor |
| B-1 | Poor |
| B-2 | Good |
| B-3 | Medium |
| B-6 | Medium |

### Crop of 1926

| Sample | Burn |
|---|---|
| C-1 | Poor |
| C-2 | Poor |
| C-3 | Medium |
| C-5 | Medium |
| C-6 | Poor |
| C-10 | Poor |
| D-1 | Poor |
| D-2 | Medium |
| D-3 | Medium |
| D-6 | Good |

Crop of 1927

| Sample | Burn |
|---|---|
| E-1 | Medium |
| E-2 | Good |
| E-5 | Good |
| E-6 | Good |
| E-9 | Poor |
| E-10 | Medium |
| F-1 | Good |
| F-2 | Good |
| F-3 | Good |
| F-6 | Good |

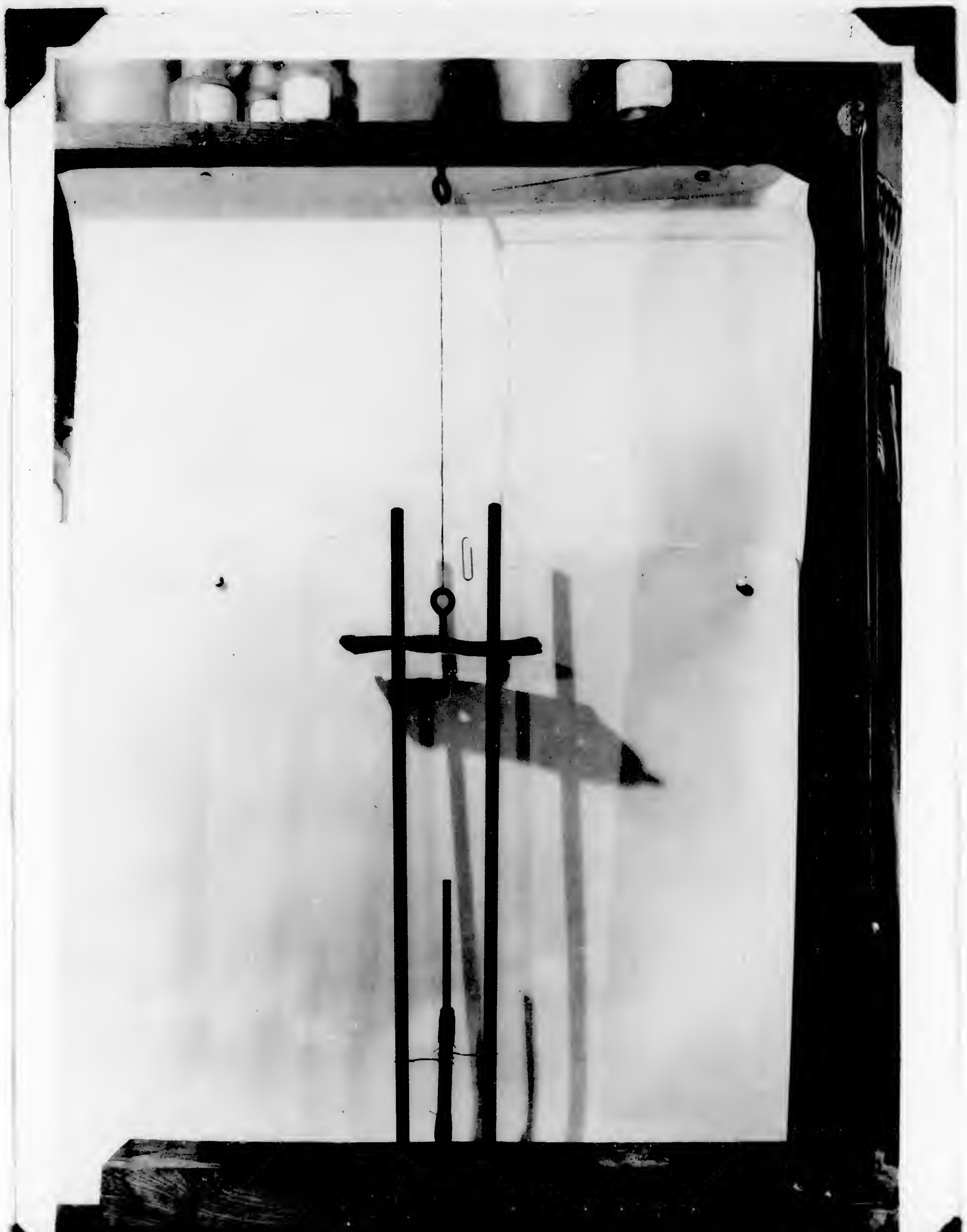

Fig.A.

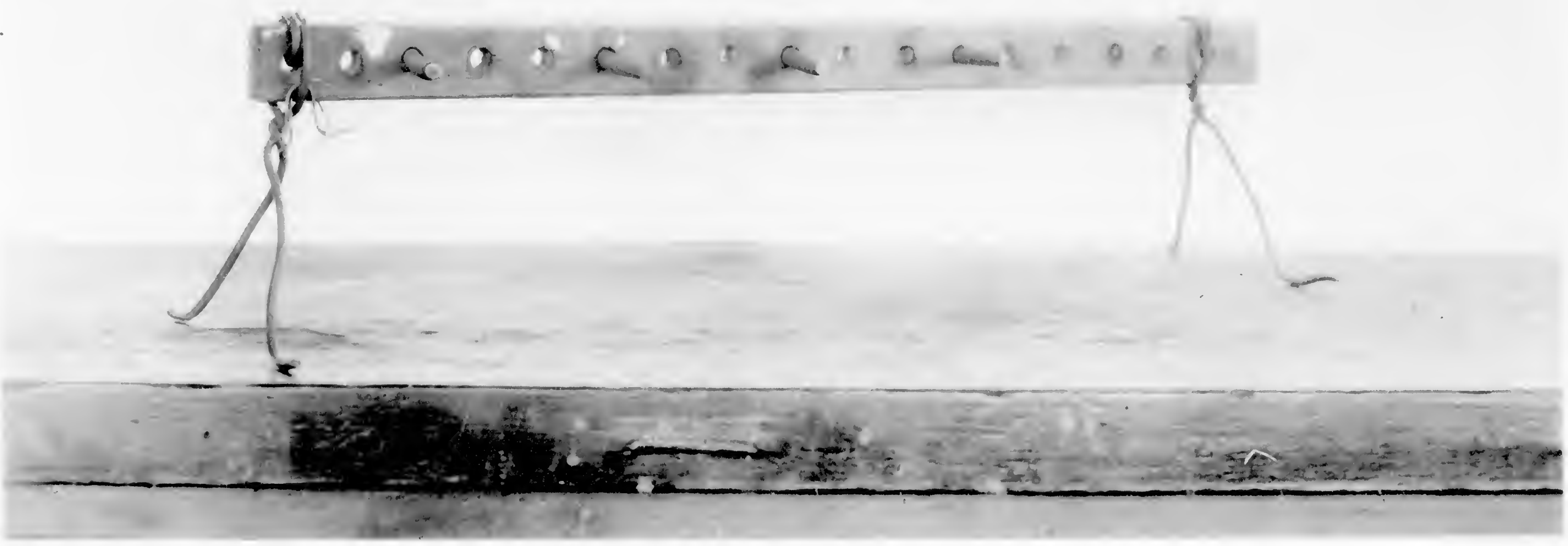

Fig.B.

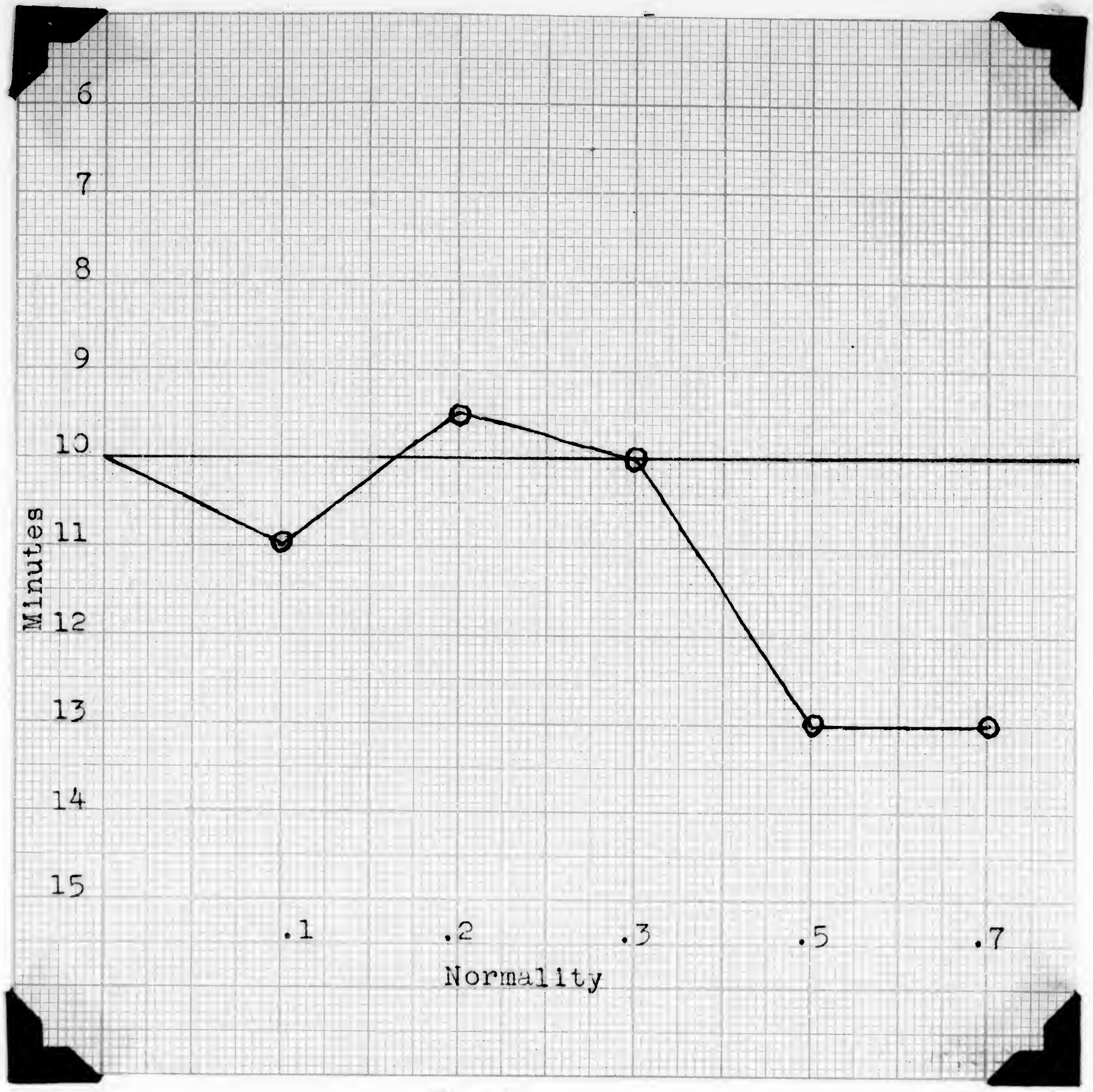

Fig. I

Showing the effect of various concentrations of potassium carbonate on the rate of combustion.

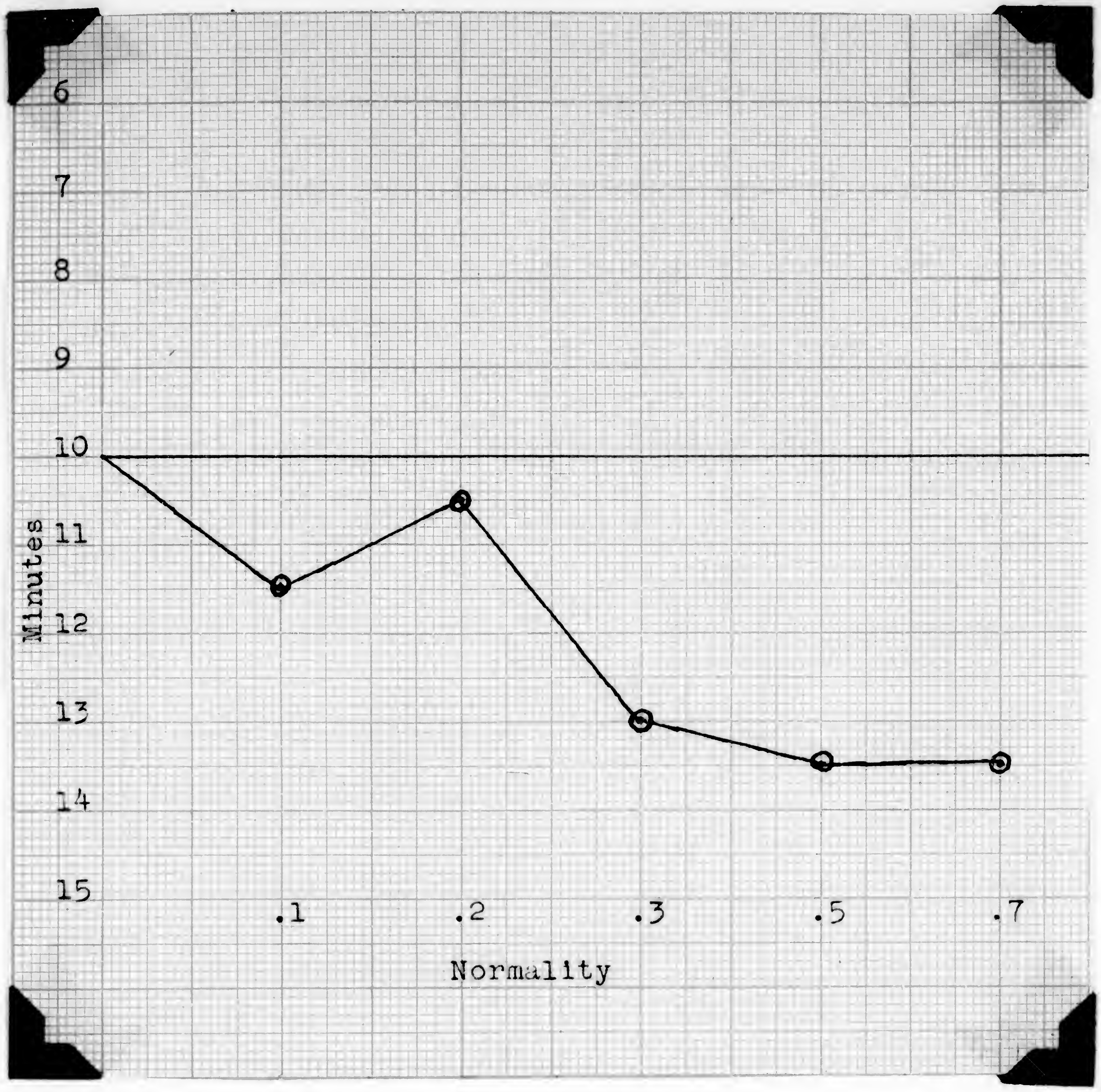

Fig. II.

Showing the effect of various concentrations of potassium hydroxide on the rate of combustion.

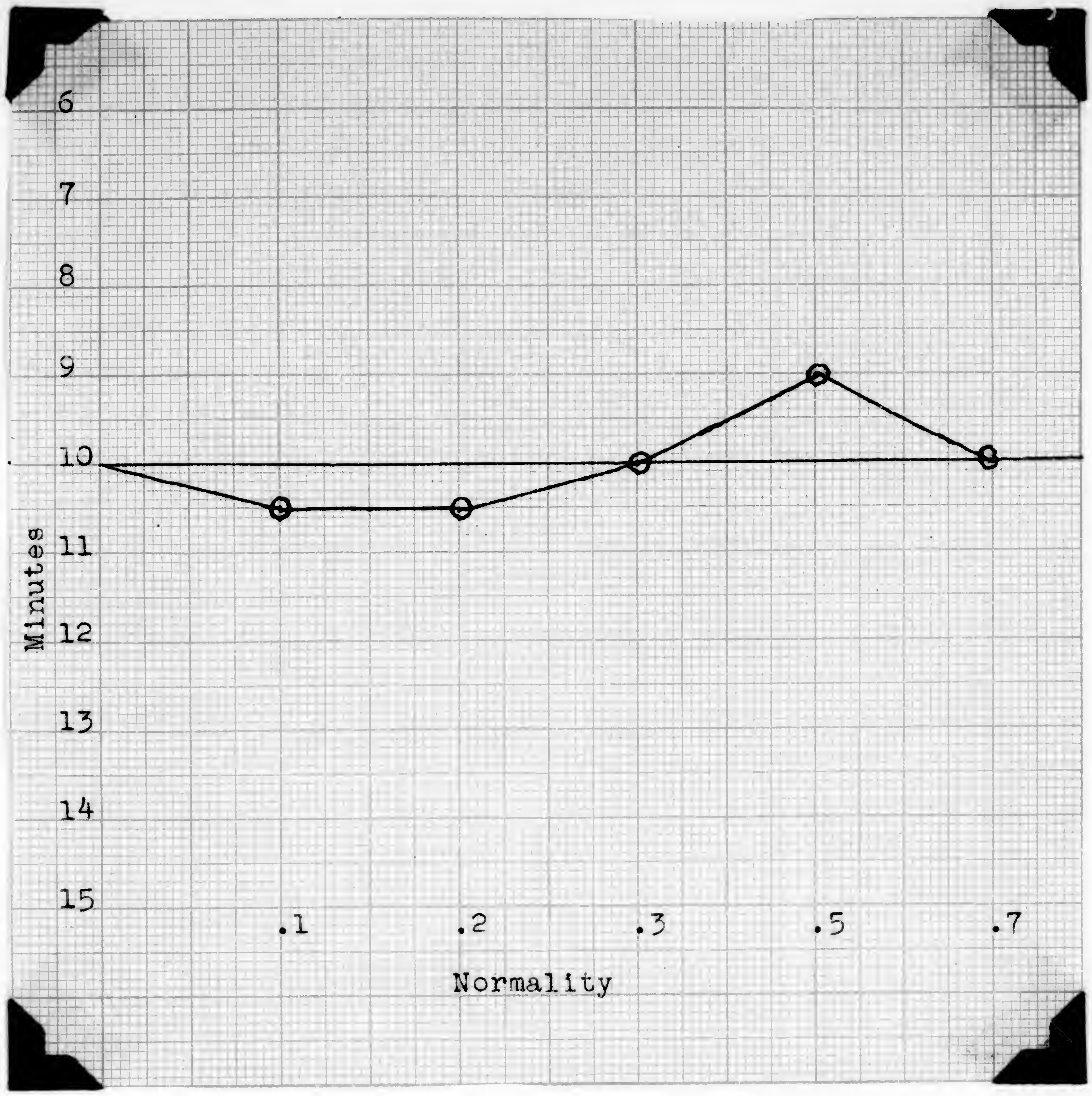

Fig.III.

Showing the effect of various concentrations of potassium citrate on the rate of combustion.

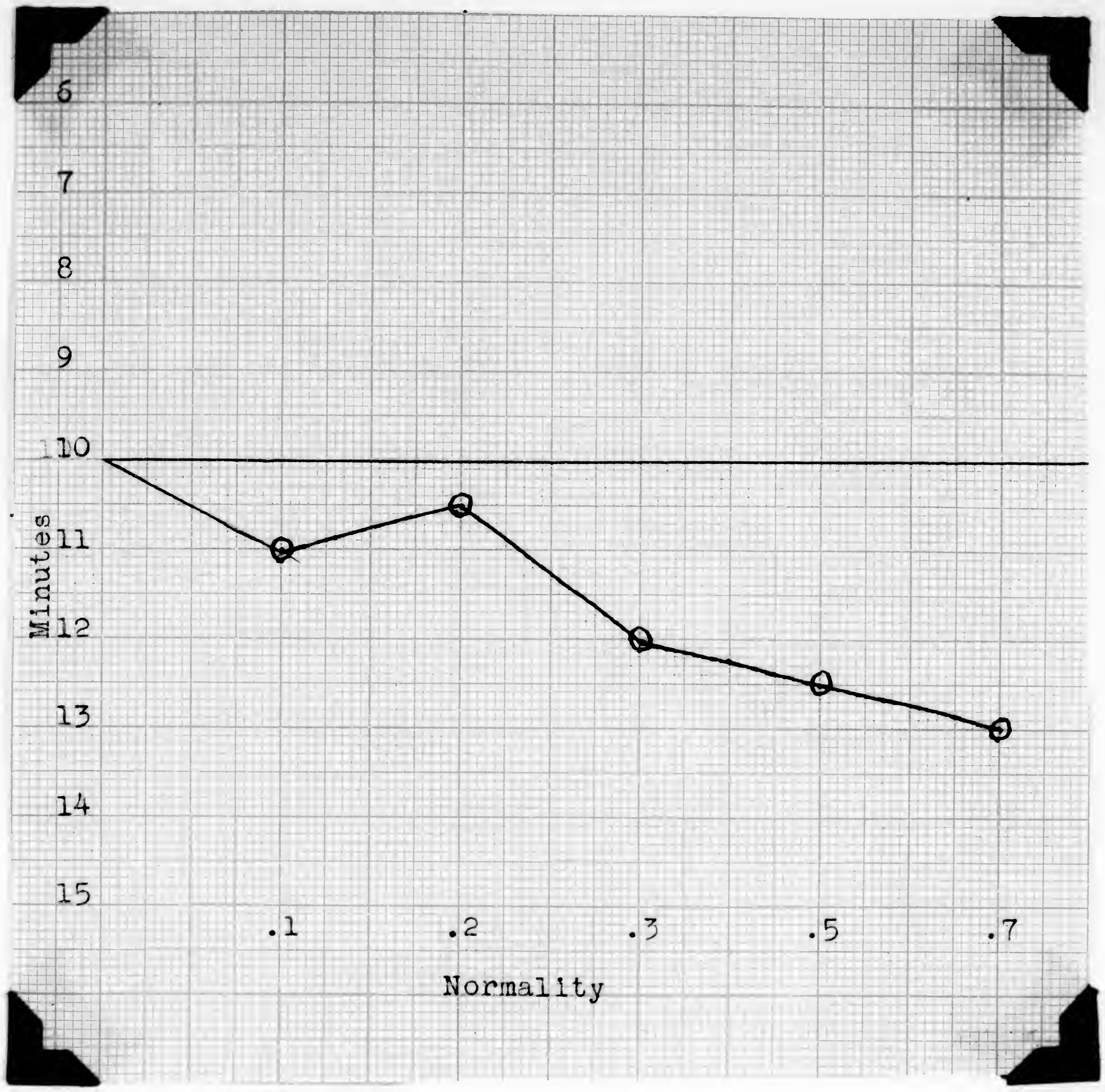

Fig. IV.

Showing the effect of various concentrations of potassium tartrate on the rate of combustion.

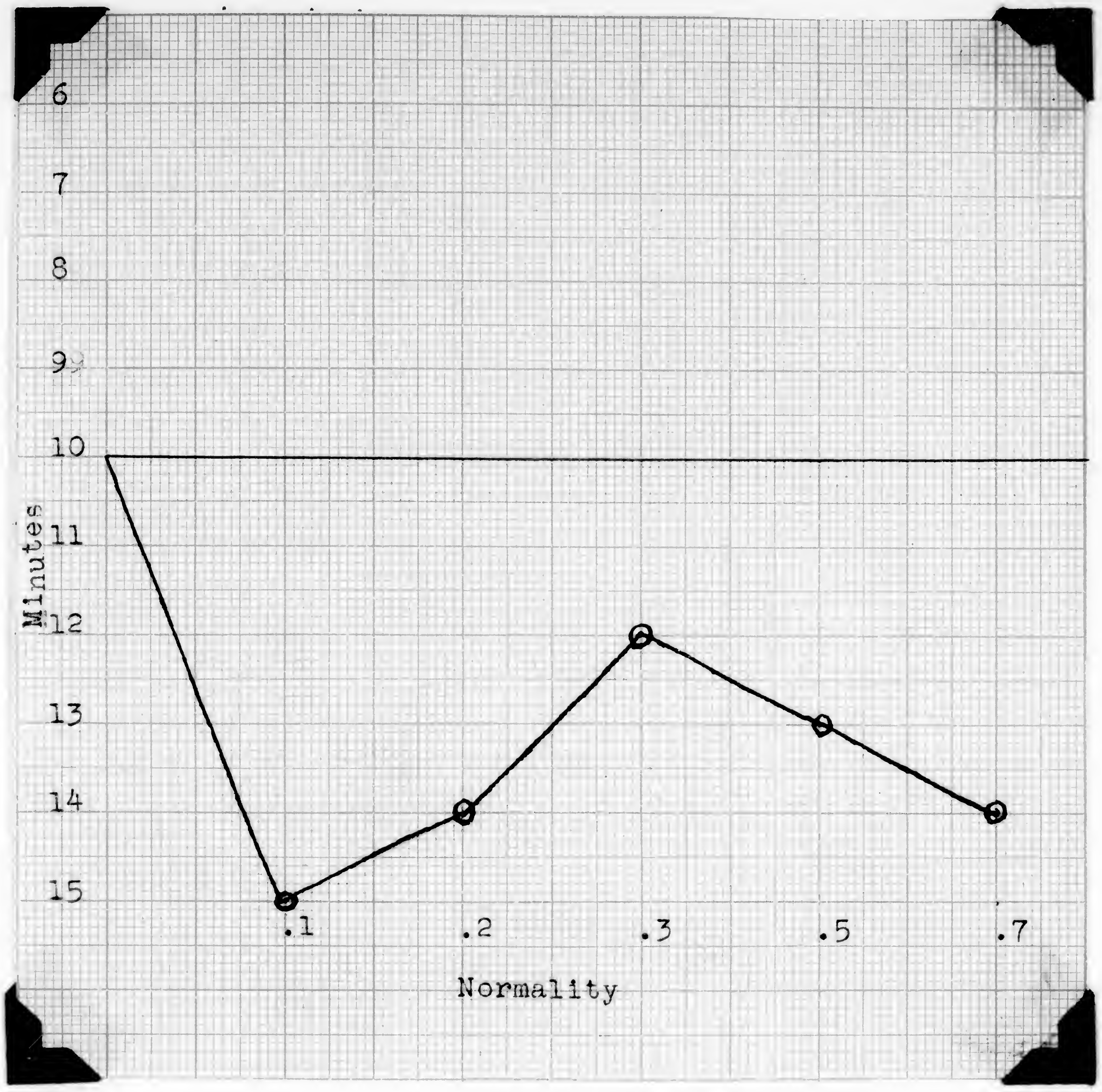

Fig. V.

Showing the effect of various concentrations of ammonium citrate on the rate of combustion.

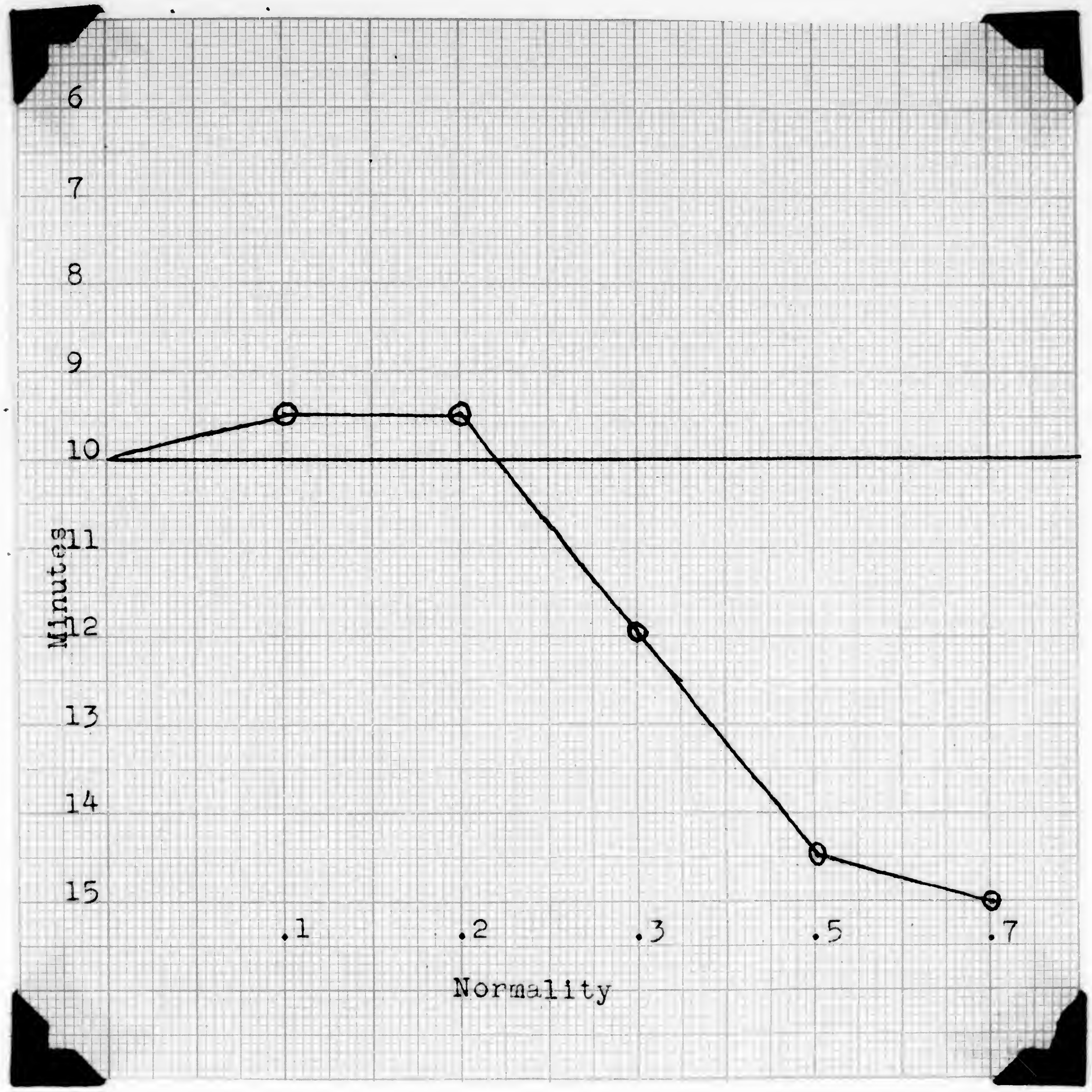

Fig.VI.

Showing the effect of various concentrations of ferric ammonium citrate on the rate of combustion.

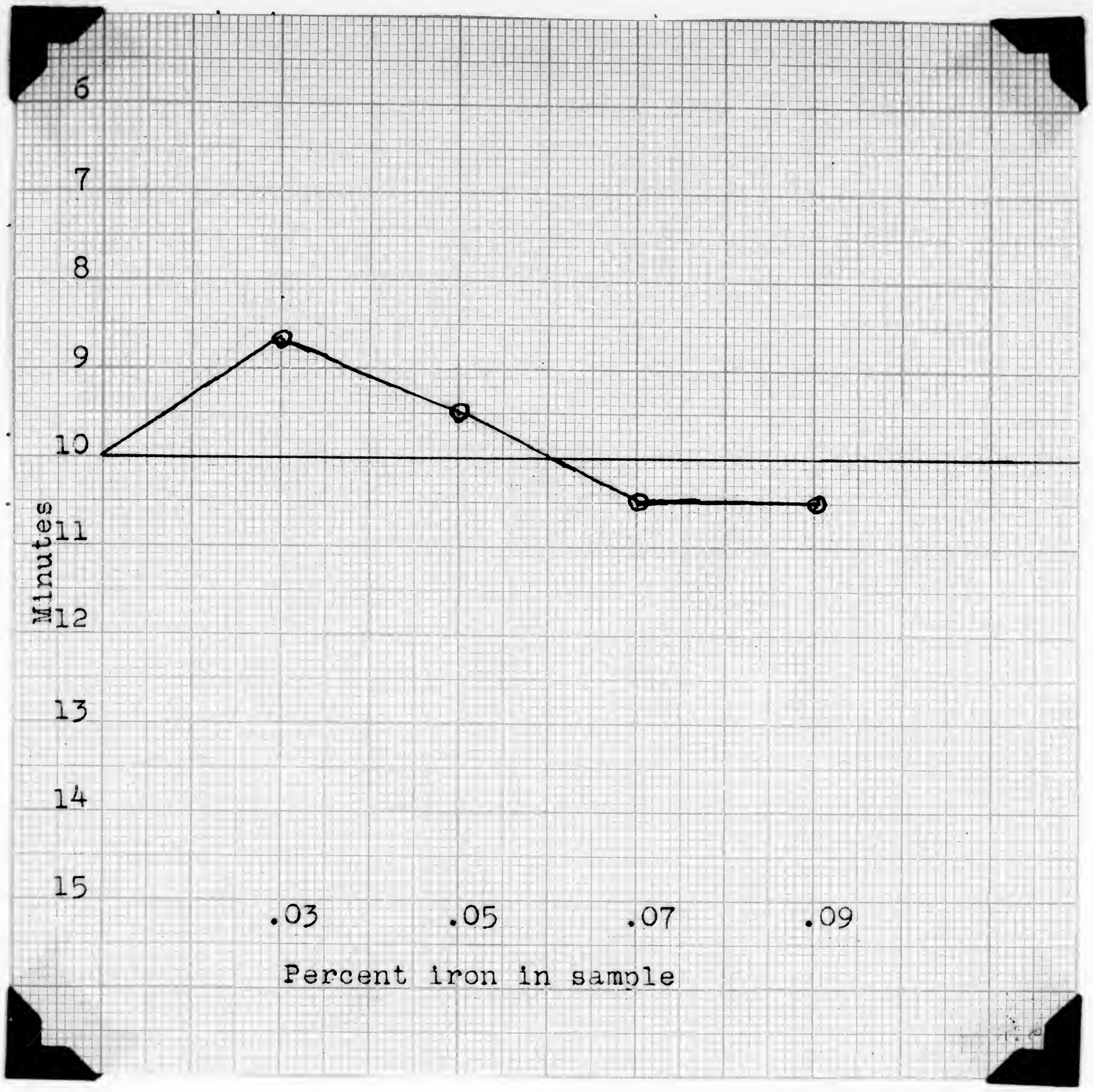

Fig. VII.

Showing the effect of .2 N potassium carbonate with varying amounts of iron on the rate of combustion.

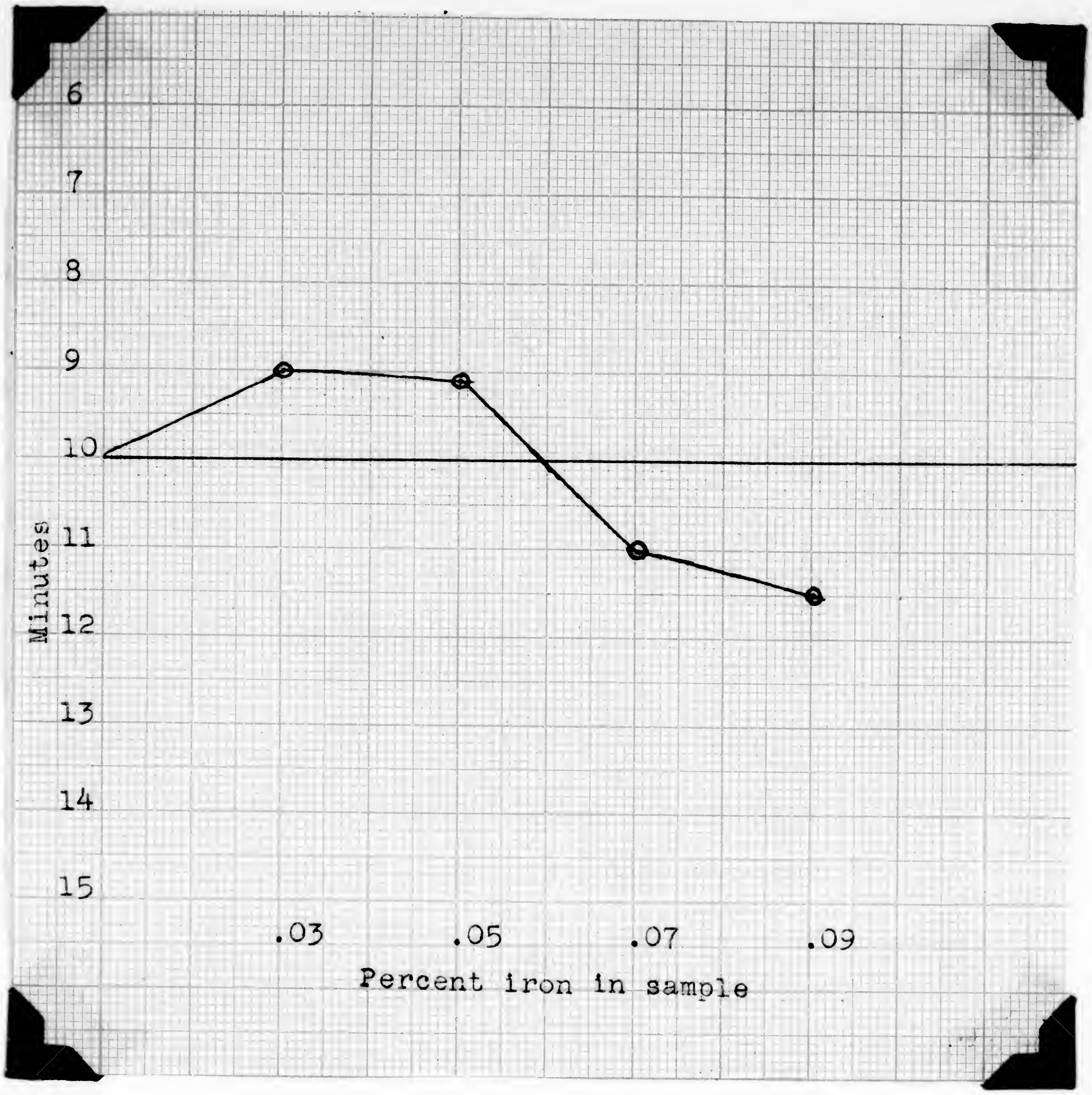

Fig. VIII.

Showing the effect of .5 N potassium citrate with varying amounts of iron on the rate of combustion.

## Discussion of Results

From the results obtained, which are partly chemical and partly physical in nature, it is apparent that the completeness and rapidity of combustion can be effected by the addition of certain chemicals.

It would seem that the effect upon the burn depends more on the nature of the combination of the element to which the catalytic effect is due rather than to the actual amount present. Of all the single salts used, potassium carbonate seems to be the most efficient.

The carbonate gives a firm cohesive ash, containing particles of carbon.

The hydroxide of potassium exerts an unfavorable influence, failing to approach the normal burn with water.

The citrate of potassium is favorable in its influence but it seems to require higher concentration to give the same effect as the .2 N potassium carbonate. It gives a firm ash which is lighter in color than that of the carbonate.

The tartrate failed to give a marked effect. No curves are plotted for the nitrate, sulfates, and chloride. While the nitrate speeds the combustion considerably, it burns with an intermittent sparkling which of course is undesirable. The sulfate and chloride failed to improve the burn, being so injurious as to completely stop combustion before the pencils had burned the required length.

It was thought that iron might possibly play an important part in improving the burn. Fig. VI shows this to be probably true.

The effect of using .2 N potassium carbonate with varying amounts of iron in the form of the acetate is shown in Fig. 7. It will be seen that the combustion is slightly better than using either the potassium carbonate or the ferric ammonium citrate alone. Almost equally good results are obtained by using .5 N potassium citrate with the iron acetate.

With varying amounts of iron and a solution of aluminium oxide no marked results were obtained, which is probably due to the precipitation of the aluminium oxide in the presence of iron.

The action of potassium in promoting the activity of iron may be due to the fact that the carbonate or citrate decomposes when heated into potassium oxide. The iron probably decomposes into its oxides, both ferrous and ferric iron being present. The potassium oxide combined with the iron oxides prevents the iron from sintering, thereby maintaining a large surface of iron. With such a large surface it is entirely possible that there is an oscillation from the ferrous to the ferric iron and vice versa. The continual supply of oxygen resulting might be the cause for the noticeable effect of these two compounds upon the combustion.

During the course of these experiments on the combustion of tobacco, it was noted that some samples had a tendency to "crack" immediately upon being ejected from the tube while others did not "crack" as readily, in fact this tendency was not evident until the samples in some cases had been dried for some time in the drying oven.

By experimentation upon the burn, it was found that those which "cracked" readily on exposure to air, invariably gave a good burn, while those which did not exhibit this tendency until later, were poor in respect to this quality.

The reason for this cracking may be due to the colloidal structure.

It is possible that the texture of the tissue itself may have its effect. It is obvious that a tough cellulose tissue would not allow swelling to a very great extent, while a softer tissue would not only allow swelling but might be of value in allowing the chemicals to penetrate the cells affording greater distribution of the materials throughout the sample.

It must be confessed that the effect of the chemicals used upon the tissue is merely a matter of speculation, but from a knowledge of permeability it is not impossible that the chemicals act as their own advance agents in changing permeability relationships so that greater distribution of the catalyzers might result.

## Summary of Results

As a result of these experiments upon the factors affecting the combustion of tobacco, it has been found that:

(1) Potassium in combination with organic acids exerts a more favorable influence on the burn than do the mineral salts of potassium.

(2) More depends on the nature of combination of potassium with other radicals than does the amount present.

(3) The rate of combustion can be accelerated by the use of iron in certain forms. Combination of the elements, iron and potassium, influences the burn to a greater extent than either one of the elements alone. The phenomenon above may be due to the promoter effect of the potassium on the iron.

(4) The structure of the tissue may have an effect on the degree of combustion.

## Acknowledgment

The writer wishes to acknowledge his appreciation of the valuable suggestions and hints made by Dr. D. E. Haley, Professor of Soil and Phytochemistry at the Pennsylvania State College, under whose direction this work was performed.

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